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U. S. NAVAL ORDNANCE LABORATORY
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VI. 5,5'-Hydrazotetrazole and the Metal Salts of 5-Nitraminotetrazole

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ABSTRACT: Neither 5,5'-Hydrazotetrazole nor the slightly soluble metal salts of 5-nitraminotetrazole are sensitive enough for use as primary explosives. Of the common metals, only the cupric, mercurous and silver salts of 5-nitraminotetrazole were slightly soluble. All of the compounds were reasonably stable in vacuum stability tests at 100°C.

Explosives Research Department
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White Oak, Maryland

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NAVORD Report 2800 4 March 1953

This report, the sixth in a series on primary explosives research, is a description of the preparation and preliminary examination of 5,5'-hydrazotetrazole and of the water insoluble salts of 5-nitraminotetrazole. Previous reports in this series are Navord Reports 2251, 2265, 2307, 2468, and 2496. It is based on work performed under Task NOL-Re2b-41-1-53. The reliability of the work and the validity of the conclusions are the responsibility of the author and of the Chemistry Division, Explosives Research Department, of the U. S. Naval Ordnance Laboratory. This report is for information only.

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Explosives Research Department
By direction

Table of Contents

Table

PRIMARY EXPLOSIVES RESEARCH

VI. 5,5'-Hydrazotetrazole and the Metal Salts of 5-Nitraminotetrazole

INTRODUCTION

The metal salts of 5-nitraminotetrazole were suggested to us as possible new primary explosives by Dr. Eugene Lieber of the U. S. Naval Ordnance Test Station, Inyokern, China Lake, California, during a visit to our laboratory. The literature reported 5,5'-hydrazotetrazole as a possible primary explosive (1). Lieber and his associates (2) have presented spectral evidence that the hydrogen on the nitramino group of 5-nitraminotetrazole is the more acidic of the two present.

Of the following metal salts of 5-nitraminotetrazole, the only ones insoluble in water were the silver, cupric, and mercurous compounds:

Cupric	Lead	Silver
Ferrous	Mercurous	Stannous
Ferric	Mercuric	Zinc
Cobalt	Cadmium	Nickel
	Beryllium	

We have been led to believe that water insolubility is a desirable property of initiating explosives, as it eliminates the possibility of reactions of the metal ions with the metal of the cups in which they are to be loaded. When the metal ions are above copper in the electromotive series some of these reactions may be avoided, but there still exists the possibility of hydrolysis and corrosion when the salt is water soluble. With a water insoluble compound, other things being equal, the effects of humidity are negligible short of substantial condensation of moisture, and the problem of hermetically sealing the cups, while still desirable, is not critical.

EXPERIMENTAL

Potassium 5-Nitraminotetrazole

A sample of potassium 5-nitraminotetrazole was prepared by the method of Lieber (3). The compound was analyzed for potassium by the method described by Pregl (4).

Calculated for CHO_2N_6K: K, 23.2

Found: K, 23.5, 23.2

Mercurous 5-Nitraminotetrazole

A 2.0 gram (0.012 moles) sample of potassium 5-nitraminotetrazole was dissolved in water and stirred mechanically. A solution containing 6.7 grams (0.024 moles) of mercurous nitrate in 75 ml of water containing 2 ml of nitric acid was added dropwise to the potassium solution. A white solid precipitated immediately. After the addition was completed the mixture was stirred an additional fifteen minutes and the solid collected on a Buchner funnel. The salt was washed with water until neutral to pH paper, sucked dry, and the drying completed under vacuum at room temperature.

The salt was analyzed for mercury by Dr. J. K. Taylor of the U. S. National Bureau of Standards, Washington, D. C. on the polarograph.

Calculated for CHO_2N_6Hg: Hg, 60.90

Found: Hg, 65.1

We were unable to find a suitable solvent for recrystallization of this sample. The physical properties observed on this sample of mercurous 5-nitraminotetrazole are listed in Table I.

Cupric 5-Nitraminotetrazole

The cupric salt of 5-nitraminotetrazole was prepared from the potassium salt using the same procedure as used in preparing the mercurous salt. The product was analyzed by the method of Pregl.

Calculated for CO_2N_6Cu: Cu, 33.3

Found: Cu, 31.2, 30.1

This salt was believed to be a hydrate, although the water of hydration was not removed by drying the sample under vacuum at 50°C for forty eight hours. If this sample is hydrated the water is held very tenaciously as was shown by the vacuum stability results. The sample had excellent thermal stability at 100°C. The physical properties observed for cupric 5-nitraminotetrazole are listed in Table I.

Silver 5-Nitraminotetrazole

The silver salt of 5-nitraminotetrazole was prepared from the potassium salt by the same procedure used in preparing mercurous 5-nitraminotetrazole. When the white solid was collected on the Buchner funnel the surface of the solid took on a purple cast. A sample was allowed to remain in the daylight and in a day's time this turned completely grey. A sample was removed from the filter immediately and dried in the dark. The dry sample was analyzed by the method of Pregl. The high silver content is believed to be due to photochemical degradation to metallic silver.

Calculated for CHO_2N_6Ag: Ag, 45.8

Found: Ag, 83.22, 83.38

This compound is believed to be too photo sensitive for use and was dropped from further consideration.

5,5'-Hydrazotetrazole

A few years ago interest was expressed in 5,5'-hydrazotetrazole by Picatinny Arsenal as a high nitrogen compound for use in propellant work (1). The compound proved to be too sensitive for their use. It was decided to prepare a sample and evaluate it as a possible primary explosive. The compound was prepared following the directions reported by Picatinny Arsenal. The physical properties observed are recorded in Table I. This compound is another example of an explosive that contains no oxygen in the molecule.

Conclusions

It is not believed that either of the three water insoluble salts of 5-nitraminotetrazole or 5,5'-hydrazotetrazole will find use as initiating explosives. None of the four compounds come close to possessing the necessary impact sensitivity for further consideration as stab initiators. Work will continue with the tetrazole nucleus in the search for new primary explosives.

Future work will be directed toward preparing derivatives of 5-amino and 1-methyl-5-aminotetrazole with the hope of finding a pure organic compound with the necessary thermal stability and impact sensitivity for serious consideration as a new initiating explosive.

ACKNOWLEDGEMENTS

The author wishes to acknowledge the assistance of Dr. J. M. Rosen for the thermal stability and ignition temperature, of B. O. Wilkerson and Dr. J. K. Taylor for metal analysis, and of G. Svadeba for the impact sensitivity measurements.

Table I

Compound	Impact Sensitivity 2.5 kg weight	Vacuum Stability cc gas/48 hrs/100°C	Hot Bar Ignition Temperature
Potassium 5-Nitraminotetrazole	21 cm	1.6 cc	
Mercurous 5-Nitraminotetrazole	38 cm	2.05 cc	256°C /1
Cupric 5-Nitraminotetrazole	68 cm	0.29 cc	256°C
Silver 5-Nitraminotetrazole	22 cm	0.24 cc	366°C
5,5'-Hydrazotetrazole	32 cm	1.7 cc	239°C

/1 All four samples detonated on the Hot Bar.

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